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# *Technical Note*

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## **ISOTOPIC ABUNDANCE RATIOS REPORTED FOR REFERENCE SAMPLES STOCKED BY THE NATIONAL BUREAU OF STANDARDS**



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**U. S. DEPARTMENT OF COMMERCE  
NATIONAL BUREAU OF STANDARDS**



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MAY 1960

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Fred L. Mohler

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# Isotopic Abundance Ratios Reported for Reference Samples

Stocked by the National Bureau of Standards

Fred L. Mohler

## Abstract

The National Bureau of Standards maintains a stock of reference samples of isotopic abundance and this paper gives results that have been reported on measurements of these samples. Some information has been received on 24 of the 32 samples. The paper includes a list of the reference samples and tables of the results reported with notes on these results.

### 1. Introduction

The National Bureau of Standards established a stock of reference samples of isotopic abundance in forms suitable for mass spectrometric analysis in 1955. [1]<sup>1</sup> The objective was to make these samples available to laboratories engaged in research on natural isotopic abundance and to serve as a clearing house for data obtained by these laboratories. In 1957 an unpublished report was prepared and distributed to interested scientists which gave a revised list of available samples and a report on measurements that had been reported to us. It is the purpose of this paper to bring this information up to date.

Table 1 lists the present stock of reference samples. This is the same as the list in the 1957 report except for the silver sample, No. 13. This was replaced by a stock of silver nitrate that had been carefully analysed for another purpose. Gas and liquid samples are distributed in break seal tubes containing about 3 ml of gas or about 0.1 g of liquid. Solid samples of about 0.1g are sealed in vacuum in Pyrex tubes.

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<sup>1</sup>Numbers in brackets refer to the Reference list at the end of the paper.

Table 1Reference Samples of Isotopic Abundance

<u>No.</u>	<u>Elements</u>	<u>Compound</u>	<u>Source</u>
1	H,O	H <sub>2</sub> O	Steam Condensate from Potomac River Water
1a	H,O	H <sub>2</sub> O	Snow Water from Yellowstone
3	D	D <sub>2</sub> O	Commercial, 99.8 atom percent D
9	Li	Li <sub>2</sub> CO <sub>3</sub>	Commercial
9a	Li	Spodumene	King's Mountain, N.C.
10	K	K <sub>2</sub> CO <sub>3</sub>	Commercial
11	Rb	Rb <sub>2</sub> CO <sub>3</sub>	Commercial
12	Sr	SrCO <sub>3</sub>	Commercial
13	Ag	AgNO <sub>3</sub>	Working standard used in a study of isotopic abundance ratio of Ag
14	N	N <sub>2</sub>	From air
15	O	Air	
20	Ca,C,O	Limestone	Solenhofen, Bavaria
21	C	Graphite	Spectroscopic grade
25	Mg	Mg(OH) <sub>2</sub>	Commercial
26	Si	SiF <sub>4</sub>	Commercial
27	B	Borax glass	Commercial
28	Si,O	Silica sand	Oriskanny deposit, Pa.
29	Cu	CuO	Commercial
100	He		Atmosphere (Commercial)
101	Ne		Atmosphere (Commercial)

(Table 1 Continued)

<u>No.</u>	<u>Elements</u>	<u>Compound</u>	<u>Source</u>
102	Ar		Atmosphere (Commercial)
103	Kr		Atmosphere (Commercial)
104	Xe		Atmosphere (Commercial)
105	Cl	NaCl	Marine, Commercial
106	Br	NaBr	Marine, Commercial
110	Cd	CdI <sub>2</sub>	Commercial
120	S	Mineral	Wharton Co., Texas
120a	S	SO <sub>2</sub>	Mineral and Air
190	Hg	Element	Density standard (limited amount) Nat. Physical Lab.
200	Pb,S	Galena	Ivigut, Greenland
200a	Pb	PbI <sub>2</sub>	From Galena
200b	Pb	Pb(CH <sub>3</sub> ) <sub>4</sub>	From Galena



## Reported Measurements of Isotope Abundances

About 220 samples have been sent to over 50 different laboratories in this country and in 14 foreign countries. Some measurements have been reported on 24 of the 32 samples listed in Table 1. It is to be noted that scientists interested in the variation of isotope abundance in nature have measured differences in abundance in different samples with a precision approaching one part in  $10^4$ . Values of abundance based solely on ion current measurements can be uncertain by as much as one part in a hundred with a sensitivity limit of about 0.01% of the maximum ion peak in conventional mass spectrometers.

Results have been reported in a variety of forms and show evidence of a wide range of precision and reliability. We have in general reported measurements in the form given to us and have not attempted to unify the method of presenting data. Numerical data are presented in Tables 2 and 3 while other pertinent information is given in the following notes. Experimental uncertainty indicated is in general given as the mean deviation except where specified. However in some instances we lack specific information on this. Numbers in parentheses are the number of measurements.

Hydrogen. The four values for D/H ratios have been corrected for mass spectrometer selectivity by comparison with standards of known D/H ratio. The differences between 1a and 1 have been reported by three laboratories. Friedman [5] finds sample 1 compared to a Lake Michigan Sample  $-0.73\%$ . Craig [6] finds the difference  $-0.79\%$ .

Deuterium. The Savannah River Plant Mass Spectrometer Laboratory measured the deuterium samples by an inter-comparison of the spectrum of  $D_2O$  with one of their standards. The absolute value is based on density measurements corrected for  $O^{17}$  and  $O^{18}$  effects. The National Bureau of Standards obtained a nearly identical value from  $D_2$  made by reduction of the  $D_2O$  but the Savannah River value is deemed more reliable.

Lithium. The result reported by Cameron [8] is based on the mass spectrum of lithium iodide. The result given by Palmer [3] was obtained with a multiple filament surface ionization mass spectrometer.

Silver. Sample 13 is from a stock of silver nitrate that has been carefully measured for another purpose. It has been measured with a multiple filament surface ionization mass spectrometer and intercompared with samples of nearly the same isotope ratio made from weighed amounts of nearly pure  $Ag^{107}$  and  $Ag^{109}$ .

Nitrogen. Palmer [3] has reported sensitive measurements of  $N^{15}$  abundance in the air sample but with the reservation that there may be some carbon monoxide interference. T. C. Hoering [12] reports that intercomparison of Sample 14 and air nitrogen collected at various times and places in the United States give  $N^{15}/N^{14}$  the same within 4 parts in 10,000. As there is no measurable variation in air nitrogen, the best values to use for Sample 14 are carefully measured published values for nitrogen. Junk and Svec [13] made a careful absolute measurement by comparison with a synthetic mixture of  $N^{15}$  and  $N^{14}$ .

Oxygen. Craig [13] has published a detailed intercomparison of oxygen in samples 1 and 20 and in air and includes values relative to various working standards of limestone. Absolute values are based on a recomputation of Nier's measurements of atmospheric oxygen. Palmer [3] reports measurements on sample 15 and we include in the table Nier's values for atomic oxygen [14] which are widely accepted as a reference standard. There is no published evidence of appreciable variations in the isotope ratios of air oxygen and the discrepancy between Palmer's values and Nier's values may be experimental uncertainty in the absolute value.

Carbon. Craig [13] and Ault [15] give comparative values for the carbon isotope ratios for various limestone working standards and sample 20 and 21a. Two of these working standards are as follows:

Nier standard limestone	$C^{13}/C^{12} \times 10^5$	1123.00	[13]
"		1124.00	[15]
Univ. of Chicago PDB standard	"	1123.72	[13]
"		1124.7	[15]

Thus Craig [13] found Nier's standard  $+0.47 \times 10^{-5}$  relative to sample 20 and Ault finds the difference  $+0.53 \times 10^{-5}$ . Gonfiantini [17] found carbon of his Carrara Marble working standard  $+3.55 \times 10^{-5}$  relative to sample 20. (The oxygen in his sample gave  $O^{16}O^{18}/O_2^{16}$ ,  $+2.55 \times 10^{-5}$  relative to sample 20). Holt [32] reported that  $C^{13}$  abundance is  $2.62 \pm 0.01\%$  less in sample 21 than in sample 20. Table 2 gives absolute values based on Craig's value for sample 20.



Silicon. Table 2 gives three measurements of the isotope abundance of Sample 26,  $\text{SiF}_4$ . David Tilles [18] has made a comparative measurement of the abundance of  $\text{Si}^{30}$  of sample 28, silica sand, and his working standard of quartz from the Mother Lode of California. The relative abundance of  $\text{Si}^{30}$  in his working standard is  $0.029 \pm 0.01$  percent heavier than sample 28. He measured  $\text{Si}^{30}/\text{Si}^{28} + \text{Si}^{29}$ .

Copper. Both the measurements reported in table 2 were made from the copper iodide mass spectrum. [19] [20]

Helium. Hoffman [21] measured the absolute abundance of  $\text{He}^3$  in sample 100 by comparison with a synthetic mixture of  $\text{He}^3$  and  $\text{He}^4$ . A sample of atmospheric helium that he prepared gave

$$\text{He}^3/\text{He}^4 = 1.37 \times 10^{-6}$$

Other Rare Gases. Some values for neon and argon are given in Table 2 and for convenience in presentation, krypton and xenon mass spectra are in Table 3. For comparison we include data published by Nier [14,22] for the rare gases. His data have been corrected for mass discrimination by comparison with carefully prepared synthetic mixtures of  $\text{A}^{36}$  and  $\text{A}^{40}$ . There is no evidence of any systematic difference between his corrected values and the other uncorrected values.

Chlorine. Parker [23] has found no significant differences in chlorine isotope abundances between sample 105 and a wide variety of natural materials.  $\text{Cl}^{37}/\text{Cl}^{35}$  values change by no more than 0.05% of the ratio. Values of the ratio for this sample have not been reported to us.

Sulfur. Bradt et al [24] used the  $\text{S}_2^+$  ions of sulfur vapor to measure the relative abundances of sulfur isotopes and they also give values based on the  $\text{SO}_2$  spectrum. Ault [15], Nief [4] and Sakai and Nagasawa [31] give values of the  $\text{S}^{34}/\text{S}^{32}$  ratio using  $\text{SO}_2$  made from sample 120 and Nief and Sakai and Negsawa also measured the value for sulfur from sample 200 (galena). Nief's values are relative to Thodes "Utah Pyrite" working standard taken as  $\text{S}^{34}/\text{S}^{32} = 4.521\%$  [25]; Ault's are relative to sulfur from Canon Diablo meteorite taken as 4.503%.

Mercury. Sample 190 was measured by four different laboratories and Dibeler has published the results [26] in a paper on "Isotope Analysis Using Dimethyl Mercury." The results will not be repeated here as the sample has not been widely distributed.

Lead. The lead samples have been carefully measured by a variety of methods. T. J. Chow [27] used a multiple filament surface ionization source to measure lead from galena. He reports the mean of 36 measurements and has applied a velocity correction to the measured currents. G. L. Bate [28] measured the spectrum of lead tetramethyl made from the lead iodide and he reports the mean of 11 sets of measurements. Dibeler [10] measured the lead tetramethyl of sample 200b and reports the mean of 11 measurements. Harman [29] and Stieff [30] have measured the lead iodide spectrum. Harman has reported the mean of 12 measurements while Stieff reported the mean of three sets of analyses two on  $Pb^{+}$  ions and one on  $PbI^{+}$  ions.

### Conclusions

We are indebted to the many scientists who have reported the measurements given in this paper. Where measurements on a sample have been reported by several laboratories the comparison gives useful information on the accuracy and reliability of isotope abundance measurements. The results add materially to the value of these samples as reference samples.

We wish to thank the U. S. Geological Survey for supplying samples 1a, 9a, 20 and 200. They also have given helpful advice on other samples. V. H. Dibeler procured the other samples and prepared the samples for distribution. He also made check measurements of isotope abundance on many of the samples as noted in the text.

Table 2. Isotopic Abundance Ratios

<u>Hydrogen</u>	<u>D/H atom %</u>	<u>Ref.</u>
Sample 1	0.0149 <sub>3</sub> ±.0001%	2.
Sample 1a	0.0128 <sub>3</sub> ±.0001%	2.
Sample 1	0.0152±.0003% Std. dev. (20 )	3
Sample 1	0.01540±.00004%	4
Sample 1a relative to Sample 1	-14.16% ± .23%	2
" "	" " -15.33%±.25%	5
" "	" " -14.25%±.25%	6
∞		
<u>Deuterium</u>		
Sample 3	99.78±0.03 atom % D	7
<u>Lithium</u>	<u>Li7/Li6</u>	
Sample 9	12.50±.05 (95% confidence level)	8
" "	12.47±.01 (97% confidence level) (52)	3
<u>Silver</u>	<u>Ag107/Ag109</u>	
Sample 13	1.0755±.0013 (95% confidence level)	9

					<u>Ref.</u>
<u>Nitrogen</u>	N <sup>14</sup>	N <sup>15</sup>			
Sample 15	99.631	0.369±.0004	Std. dev.(20)		3.
	99.634	0.3662±.0004			16.
<u>Oxygen</u>	O <sup>16</sup> O <sup>18</sup> /O <sub>2</sub> <sup>16</sup>				
Sample 1	412.44 x 10 <sup>-5</sup>				13.
Sample 20	414.08 x 10 <sup>-5</sup>				13
Air O <sub>2</sub>	409.40 x 10 <sup>-5</sup>				13
	O <sup>16</sup>	O <sup>17</sup>	O <sup>18</sup>		
Sample 15	99.755	0.0387±.0004	0.2066±.0002 Std. dev. (20)		3
Air	99.759	0.0374	0.2039		14
<u>Carbon</u>	C <sup>13</sup> /C <sup>12</sup>				
Sample 20	1122.53 x 10 <sup>-5</sup>				13
	1123.47 x 10 <sup>-5</sup>				15
Sample 21	1092.49 x 10 <sup>-5</sup>				13
	1092.9 x 10 <sup>-5</sup>				15
	1093.1 x 10 <sup>-5</sup>	(Based on Sample 20- 1122.5 x 10 <sup>-5</sup> )			32

	Si <sup>28</sup>	Si <sup>29</sup>	Si <sup>30</sup>	Ref.
<u>Silicon</u>				
Sample 26	92.20	4.685±.002	3.117±.002 Std. dev. (20)	4.
	92.15	4.72±.02	3.13±.02	11
	92.19	4.69±.02	3.12±.01 (4)	10
<u>Copper</u>				
	Cu <sup>63</sup> /Cu <sup>65</sup>			
Sample 29	2.223±.001	95% Confidence level		19
	2.230±.002	"	"	20
<u>Helium</u>				
	He <sup>3</sup> /He <sup>4</sup>			
Sample 100	1.341±.03 x 10 <sup>-6</sup>			21
<u>Neon</u>				
	20	21	22	
Sample 101	90.65	0.265±.001	9.084±.01 (24)	10.
"	90.82	.23	8.93 (2)	11.
Air Neon	90.92±.04	.259±.001	8.82±.04	22.
<u>Argon</u>				
	36	38	40	
Sample 102	0.346±.002	0.069±.001	99.585 (24)	10
"	0.33	0.05	99.62 (2)	11
Air argon	0.337±.001	0.0630±.0001	99.600	14



<u>Sulfur</u>	32	33	34	36	
Sample 120	95.0	0.760±.004	4.22±.01	0.014 (5)	24
Sample 120a	95.0	0.772±.01	4.23±.07	- (6)	24
	s <sup>34</sup> /s <sup>32</sup>				
Sample 120	4.509±.003%	95% confidence level			15
120	4.487±.008%				4
120,120a	4.494±.009%				31
200	4.506±.017%				4
200	4.493±.009%				31
<u>Lead</u>	204	206	207	208	
Sample 200	1.540±.002	22.48±.01	22.62±.01	53.36±.02	27
Sample 200a	1.53±.006	22.60±.04	22.56±.02	53.31±.03	28.
Sample 200b	1.53±.02	22.63±.17	22.68±.21	53.13±.19	10.
Sample 200a	1.54±.01	22.49±.03	22.68±.03	53.29±.05	29
Sample 200a	1.539±.003	22.530±.04	22.636±.02	53.293±.06	30

Table 3Krypton and Xenon Isotope Abundances

Krypton, Sample 103 and Univ. of Minn. Sample				
m/e	<u>Ref. 4*</u>	<u>Ref. 10**</u>	<u>Ref. 11***</u>	<u>Ref. 22</u>
78	0.351±.001	0.354±.001	.355	0.354±.002
80	2.256±.005	2.28±.03	2.28	2.27±01
82	11.52±.02	11.66 <sub>2</sub> ±.03	11.6	11.56±.02
83	11.49±.015	11.578±.04	11.56	11.55±.02
84	56.99±.06	56.77±.04	56.92	56.90±.1
86	17.41±.03	17.35±.02	17.25	17.37±.02
Xenon, Sample 104 and Univ. of Minn. Sample				
m/e	<u>Ref. 4*</u>	<u>Ref. 10**</u>	<u>Ref. 11***</u>	<u>Ref. 22</u>
124	0.094±.001	0.094±.001	(.10)	0.096±.001
126	0.088±.001	0.090±.001	(.10)	0.090±.001
128	1.921±.007	1.923±.006	1.93 <sub>5</sub>	1.919±.004
129	26.25±.05	26.33±.04	26.39	26.44±.08
130	4.10±.01	4.135±.01	4.15 <sub>5</sub>	4.08±.01
131	21.16±.05	21.19±.03	21.15	21.18±.05
132	26.91±.04	26.93±.02	26.89	26.89±.07
134	10.49±.03	10.41±.02	10.48	10.44±.02
136	8.94±.02	8.87±.03	8.83 <sub>5</sub>	8.87±.01

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\*Standard deviation in over 20 measurements.

\*\*Mean of 24 measurements of krypton and 11 of xenon.

\*\*\*2 measurements.



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## NATIONAL BUREAU OF STANDARDS

A. V. Astin, *Director*

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